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Eweka

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(54) **PREPARATION OF ELECTRODE COMPOSITIONS**

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See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

3,932,586 A 1/1976 Guerrieri

4,247,525 A 1/1981 Voeste

(Continued)

FOREIGN PATENT DOCUMENTS

DE 4015147 7/1991

GB 2266179 10/1993

(Continued)

OTHER PUBLICATIONS

Friend et al., "Lithium Sulphite and Some Derivatives", Journal of the Chemical Society, Jan. 1, 1928, pp. 2245-2248.

(Continued)

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(57)

ABSTRACT

The invention relates to processes for the preparation of electrode compositions, especially those intended for use in supercapacitors. A process is provided for preparing lithium sulphite comprising the steps of:—a) introducing H_2SO_3 (aq) into a reaction vessel; b) reacting the H_2SO_3 (aq) with an aqueous suspension of Li_2CO_3 in the vessel to form an aqueous solution of $\text{Li}_2\text{—CO}_3$; and c) evaporating the solution to recover Li_2CO_3 (s), wherein at least steps a) and b) are conducted under an inert atmosphere. Preferably, in step b) H_2SO_3 (aq) and Li_2CO_3 (aq) are reacted with each other in substantially equimolar amounts. There is also provided a process for forming an electrode material comprising a complexing step of causing lithium sulphite to form SO_3 complexes at active N sites of a nitrogen-carbon structure, in the presence of a selected amount of a sink that absorbs the liberated lithium, so as to form the N:SO_3 complexed electrode material. Preferably, the nitrogen-carbon structure is thermally restructured polyacrylonitrile (TR-PAN) or a copolymer thereof.

10 Claims, 6 Drawing Sheets

